

PATENT SPECIFICATION

723.598



Date of Application and filing Complete Specification: Sept. 4, 1952.

No. 22267/52.

Application made in Netherlands on Sept. 7, 1951.

Complete Specification Published: Feb. 9, 1955.

Index at acceptance:—Classes 2(5), P2A, P2C8(A: B: C), P2C20A, P2C20D(1: 3), P2(D1A: K8: S2), P2T(1X: 2A), P7A, P7C8(A: C), P7(D1A1: S2: T1X), P8A, P8C8(A: B: C), P8C20A, P8C20D(1: 3), P8D(2B2: 3A), P8(K2: S2), P8T(1X: 2A), P10A, P10C8(A: C), P10(D1A: S2: T1X), R1C(1: 6), R2C(1: 6), R21C(1: 6); and 82(2), F1(B1B: X), F2(H: U: Y), F4N.

COMPLETE SPECIFICATION

Improvements in or relating to methods of producing Electrically Conductive Mouldings from Plastics

We, N. V. PHILLIPS' GLOEILAMPEN-FABRIEKEN, a limited liability Company, organized and established under the laws of the Kingdom of the Netherlands, of Emmasingel 29, Eindhoven, Holland, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the manufacture of electrically conductive articles from plastics.

If conductive articles are to be produced in a known manner by moulding plastics i.e. polymerisation and condensation products whether or not capable of being hardened, it is necessary to add to the plastics a considerable quantity of conductive filler, for example metal powder or carbon. In order to obtain a satisfactory conductivity, the conductive material should amount to approximately 50% by weight or more of the total mass. Due to this, the mechanical properties of such mouldings, in comparison with mouldings without conductive filler, will be far worse.

In accordance with the invention, conductive mouldings having satisfactory mechanical properties are obtained by reducing plastics to grains, the individual grains being provided with electrically conductive coatings and the powder thus obtained being subsequently pressed to mouldings while heating.

In this case, deformation of the plastic grains and consequently also of the conductive coatings occurs, but in spite thereof a continuous network of conductive layers is obtained, which is comparable to a honeycomb. The bond between the grains is established, it may be assumed, by the plastics as a result of slight damage to the conductive coatings caused during the moulding operation.

This method has the special advantage that, averaged over the whole mass, a quantity of conductive material of only a few per cent. is sufficient.

The conductivity of products according to the invention is controllable by the choice of the size of grain of the plastics, by the thickness of the conductive coatings on the grains, by the choice of the conductive material and of the temperature, pressure and time in moulding.

Excellent results are obtained if the plastics are pulverized to grains of 0.3 mm and smaller. The plastics may, for example, be reduced to grains by powdering or cutting, or again by drying drops of a solution. Alternatively granular plastics obtained by suspension polymerisation may be used.

As conductive materials use is preferably made of metals such as silver and copper, or of carbon.

The conductive layers may be applied in many different manners onto the plastic grains. Silver, copper and nickel may, for example, be precipitated from a solution by means of a reducing agent. As an alternative, the conductive layer may be obtained by precipitating the conductive material from a suspension. Sometimes, for example in the case of carbon powder and also in the case of copper powder, obtained by reduction of copper compounds, a conductive layer may be obtained on the grains by carefully rubbing a mixture of the powders together in a dry state. Further, in the case of carbon, by mixing grains of plastic with a suspension of graphite the grains of plastic may be coated with a layer of graphite. The rubbing of carbon and copper powders with plastic grains may be carried out, for example, in a ball mill. In this case it is important that hard and heavy balls which would grind the mix-

ture are not used. Only balls such as wooden balls which are soft and light may be used.

In order that the invention may be readily carried into effect, a number of examples will now be described in detail.

EXAMPLE I.

15 gms. of polystyrene in the form of a suspension-polymer with a grain diameter of approximately 0.04 mm. are treated with a mixture of 20 ccs. of a silvering solution (17.1 gms. of AgNO_3 , 8.6 gms. of NaOH and 48 ccs. of concentrated ammonia per litre) and 1 ccs. of a reducing liquid (190 gms. cane sugar, 370 ccs. of alcohol of 96% and 3 ccs. of concentrated nitric acid per litre). After two hours the silvered polystyrene grains are separated by filtering and washed with water. A rod moulded from this material at 170°C . and under a pressure of 100 kgms/cm² had a specific resistance of $2\Omega\text{cm}$.

EXAMPLE II.

For coppering 4 gms. of polymethylacrylic methylester with a grain diameter of 15-microns, use is made of a mixture of three solutions A, B and C in quantities of 13 ccs., 5.3 ccs. and 10 ccs. respectively. The composition of solution A is 50 gms. of copper acetate, 200 ccs. of concentrated ammonia, 260 ccs. of distilled water; that of solution B is 17.5 gms. of KOH , 110 ccs. of distilled water; that of solution C is 15 ccs. of hydrazine hydrate, 39 ccs. of distilled water. The copper is precipitated on the grains of the polymer by treatment with the mixture of the solutions for two hours while heating on a water-bath. A rod obtained from the copper-plated grains by moulding at 170°C . and under a pressure of 100 kgms/cm² had a specific resistance of $140\Omega\text{cm}$.

EXAMPLE III.

Polymer grains are nickel-plated with the use of a mixture of three solutions A, B and C in quantities of 30 ccs., 30 ccs. and 6 ccs. respectively. The composition of solution A is 50 gms. of nickel acetate, 200 ccs. of concentrated ammonia, 360 ccs. of distilled water. Solution B is a 28% solution of hydrazine hydrate solution, solution C being a 0.16% solution of potassium platinum chloride which acts as a catalyst. 5 gms. of powdered polystyrene with a grain size of approximately 0.04 mm. are treated for one hour at 75°C . with the mixture of the solutions. A rod moulded at 170°C . and under a pressure of 100 kgms/cm² had a specific resistance of approximately $0.5\Omega\text{cm}$.

EXAMPLE IV.

30 gms. of granular polystyrene are carefully rubbed together with 5 gms. of graphite powder for half an hour while heating to 150°C ., thus covering the polystyrene grains with layers of graphite. Any graphite not

bound may be removed by sieving out or by shaking out with water. Analysis proved that the carbon covered polystyrene grains contained only 1.5% of graphite. Moulding at a temperature of 170°C . and under a pressure of 100 kgms/cm² yielded a rod having a specific resistance of $54\Omega\text{cm}$.

EXAMPLE V.

20 gms. of an ureaformaldehyde moulding powder with a grain diameter smaller than 0.15 mm. are carefully rubbed together for one hour with 600 mgms. of carbon powder in a ball mill with wooden balls, substantially the whole quantity of carbon being adsorbed at the surface of the grains. A rod moulded at 150°C . and under a pressure of 1000 kgms/cm² had a specific resistance of $150\Omega\text{cm}$.

EXAMPLE VI.

25 gms. of a granular synthetic rubber mixture having a composition of 200 gms. of a copolymer of butadiene with acrylonitrile, 10 gms. of zinc oxide, 3 gms. of tetramethyl thiuram disulphide, 2.5 gms. of sulphur, 1 gm. of stearic acid, 50 gms. of dibutyl phthalate are carefully rubbed together with 2.5 gms. graphite powder in a ball mill with wooden balls. Vulcanisation of the carbon coated grains under a pressure of 100 kgms/cm² at 150°C . for 20 minutes yielded an elastic conductive product having a specific resistance of $2000\Omega\text{cm}$.

EXAMPLE VII.

Granular polyvinyl chloride having a content of plasticiser of 50% averaged on polyvinyl chloride is rubbed carefully with 10% of colloidal graphite. After moulding at 150°C . and under a pressure of 100 kgms/cm² the carbon coated grains yielded a conductive product having a specific resistance of $6000\Omega\text{cm}$.

EXAMPLE VIII.

Granular alkaline cresol resin obtained by the partial condensation of cresol with an aldehyde in an alkaline solution, to which resin is added a hardening agent e.g., hexamethylene tetramine, is coated with carbon by carefully rubbing the resin and graphite together in a ball mill with wooden balls. The carbon coated resin grains are then pressed under a pressure of 1000 kgms/cm² for five minutes at 170°C . to form plates 2 mm. to 2.5 mm. thick. In order to gain an idea of the influence exerted by the size of grain on the value of the resistance the latter was measured on plates having a surface of 0.8 cm² and obtained from grains of different size and slightly varying carbon content. Moreover, several mechanical characteristics were measured. The results of these measurements are stated in the following table.

	Grain Diameter in mm.	% carbon	Resistance in Ω	Impact Strength kgcm/cm ²	Impact Bending kgcm/cm ²	Bending Strength kg/cm
	0.3 to 0.6	9	8	0.69	2.6	310
5	0.3 to 0.21	9.9	47	0.69	2.6	310
	smaller than 0.21	12.5	200	1.2	7.0	510

What we claim is:—

1. A method of producing electrically conductive mouldings from plastics, characterized in that plastics are reduced to grains, the individual grains being provided with conductive coatings and the powder thus obtained being subsequently pressed to mouldings while heating.
- 10 2. Moulding produced by the method claim

in claim 1.

3. A method of producing electrically conductive mouldings substantially as described in any one of the examples given herein.

T. D. THREADGOLD,
Chartered Patent Agents,
Century House, Shaftesbury Avenue,
London, W.C.2,
Agent for the Applicants.

This document is printed for Her Majesty's Stationery Office, by the Courier Press.—1955.
Copies may be obtained from the Patent Office, 25, Southampton Buildings, London, W.C.2, from which

**PUBLISHED BY :—
THE PATENT OFFICE,
25, SOUTHAMPTON BUILDINGS,
LONDON, W.C.2.**

BEST AVAILABLE COPY

This Page Blank (uspto)